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(54) LITHIUM NICKEL MANGANESE OXIDE, ITS MANUFACTURING METHOD AND LITHIUM-ION SECONDARY CELL USING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide $LiNi_{1/2+a}Mn_{1/2-a}O_2$ which suppresses cation mixing between the 3a-3b sites significant in Li Ni Mn O and has excellent output under high load and to provide a method for manufacturing the same.

SOLUTION: The compound is a positive electrode active substance expressed by the general formula of $Li_{1+x}Ni_{1/2+a}Mn_{1/2-a}O_2$ with x and a satisfying $x \geq 0$ and $-0.05 \leq a \leq 0.05$ and shows ≥1.15 ratio of diffraction peak intensities (003)/(104) on the (003) face and the (104) face in terms of the Miller indices hkl in the powder X-ray diffraction using Cu-Ka ray. The method for manufacturing the compound includes a process of mixing nickel manganese oxide having an illmenite structure with a lithium compound and a process of calcining the mixture at ≥750° C in an oxygen-containing atmosphere.

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CLAIMS

[Claim(s)]
[Claim 1]
General formula Li1+XNi1/2+alphaMn1/2-alpha It is the lithium-nickel-manganese multiple oxide which sets to X>=0 X value in the formula expressed with O2, and sets alpha value to -0.05<=alpha<=0.05. The twin crystal particle which makes a twin plane the field in Miller indices hkl (104) to which the particle of said multiple oxide is hexagonal and belongs is contained. Diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) of the powder X diffraction using Cu-K alpha rays is 1.15 or more. And after dissolving said 0.3g of lithium-nickel-manganese multiple oxides and 3.0g of potassium iodide in the condition of having shaded to 6 N-HCl50cc, The lithium-nickel-manganese multiple oxide whose average valence of the nickel and manganese which were measured by the iodine titrimetric method which titrates by 0.1 N-Na2S2O3 after adding 1 N-NaOH200cc and one cc of starch solutions is 3.1 or less [3.0 or more].
[Claim 2]
General formula Li1+XNi1/2+alphaMn1/2-alpha Lithium-nickel-manganese multiple oxide according to claim 1 with which X value in the formula expressed with O2 sets 0.01<=X<=0.10 and alpha value to -0.05<=alpha<=0.05.
[Claim 3]
The manufacture approach of the lithium-nickel-manganese multiple oxide according to claim 1 or 2 characterized by calcinating the process which mixes an ilmenite structured type nickel-manganic acid ghost and a lithium compound, and said mixture with the burning temperature of 750 degrees C or more under an owner oxygen ambient atmosphere.
[Claim 4]
The manufacture approach of the lithium-nickel-manganese multiple oxide according to claim 3 characterized by calcinating the process which mixes an ilmenite structured type nickel-manganic acid ghost and a lithium compound, and said mixture under an owner oxygen ambient atmosphere with 750-degree-C or more burning temperature of 950 degrees C or less.
[Claim 5]
The manufacture approach of the lithium-nickel-manganese multiple oxide according to claim 4 which is an oxide obtained when the average oxidation number of nickel and manganese compounded because an ilmenite structured type nickel-manganic acid ghost oxidizes a nickel-manganese compound hydroxide calcinates 2.5 or more nickel-manganese compounds under an owner oxygen ambient atmosphere at 300 degrees C or more less than 800 degrees C.
[Claim 6]
The manufacture approach of a lithium-nickel-manganese multiple oxide according to claim 5 that burning temperature is 300 degrees C or more 500 degrees C or less.
[Claim 7]
The manufacture approach of the lithium-nickel-manganese multiple oxide according to claim 5 which compounds a nickel-manganese compound hydroxide under complexing agent coexistence.

[Claim 8]

The manufacture approach of a lithium-nickel-manganese multiple oxide according to claim 7 that a complexing agent is ammonia.

[Claim 9]

The manufacture approach of the lithium-nickel-manganese multiple oxide according to claim 3 characterized by calcinating the process which mixes an ilmenite structured type nickel-manganic acid ghost and a lithium compound, and said mixture under an owner oxygen ambient atmosphere with 900-degree-C or more burning temperature of 1050 degrees C or less.

[Claim 10]

The manufacture approach of the lithium-nickel-manganese conjugated compound according to claim 9 which is a nickel-manganic acid ghost which said ilmenite structure nickel-manganic acid ghost calcinated the nickel-manganese compound expressed with general formula nickel1/2+alphaMn1/2-alpha(OH)y(COO) z-nH2O (y+z=2, 0.5<=z<=1.5, and alpha value -0.05<=alpha<=0.05, n>=0) under the owner oxygen ambient atmosphere, and was obtained.

[Claim 11]

The manufacture approach of a lithium-nickel-manganese multiple oxide according to claim 10 that burning temperature is 550 degrees C or more less than 800 degrees C.

[Claim 12]

Positive active material for rechargeable lithium-ion batteries containing a lithium-nickel-manganese multiple oxide according to claim 1 or 2.

[Claim 13]

The rechargeable lithium-ion battery which uses positive active material according to claim 12.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]
This invention relates to the lithium-nickel-manganese multiple oxide used for the positive active material for rechargeable batteries etc., its manufacture approach, and the rechargeable lithium-ion battery using it.
[0002]
[Description of the Prior Art]
The miniaturization of an AV equipment, a cellular phone, a personal computer, etc. and cordless-ization are progressing quickly in recent years, and $LiCoO_2$, $LiNiO_2$, $LiMn_2O_4$, etc. have been studied as these power sources for a drive. However, all have some which are hard to be called capacity, safety, and ingredient with which are satisfied of all costs, and, recently, a Li-nickel-Mn multiple oxide like $LiNi_{0.5}Mn_{0.5}O_2$ is expected as an ingredient with which are satisfied of a high energy consistency, safety, and cost. (For example, nonpatent literature 1 reference)
Conventionally, many dry-blending methods (for example, nonpatent literature 2 reference) which mix powder, such as NiO, MnO, and $LiOH-H_2O$, and are acquired have been used for composition of such a Li-nickel-Mn multiple oxide. However, when Mn content is high, since atomic homogeneity is inadequate, an impurity phase is contained and it did not come to acquire electrochemistry property that crystallinity is low and sufficient.
[0003]
Moreover, in the approach using the wet slurry which makes powder, such as Mn oxide, nickel solution, Li solution, etc. the shape of a slurry, and is mixed and calcinated, although it compares with dry process and an improvement is found in respect of crystallinity, an electrochemistry property, etc., it has technical problems, such as generating of corrosive gas, such as nitrogen oxides.
[0004]
Improvement in the homogeneity dispersibility of a configuration atom is possible by on the other hand carrying out coprecipitation with gestalten, such as a hydroxide, a carbonate, and an oxalate, in a coprecipitation method. In a coprecipitate, since the crystal structure of a precursor also influences greatly reactivity with a lithium compound as a result the crystal structure of a Li-nickel-Mn multiple oxide, and the cell engine performance, selection of a precursor becomes important. For example, by the approach of an indication, although the method of obtaining a direct Li-nickel-Mn multiple oxide is indicated from the nickel-manganese compound hydroxide and the lithium compound as an approach of obtaining a Li-nickel-Mn multiple oxide, since the manganese divalent hydroxide is unstable, a problem may arise in the homogeneity of a precursor in actuation in atmospheric air. (For example, patent reference 1 reference) Therefore, the homogeneity dispersibility of a configuration atom was high and establishment of selection of the nickel-Mn compound which is rich in reactivity with a lithium compound, and has the stable crystal structure in atmospheric air, and the manufacture approach of the Li-nickel-Mn compound using the nickel-Mn compound was pressing need.

[0005]
Moreover, conventionally, baking in the elevated temperature of 1000 degrees C or more is made desirable, and composition of $LiNi0.5Mn 0.5O2$ which used the nickel-Mn compound hydroxide for the raw material also has a report that a problem is in a cycle life below 850 degrees C. (For example, patent reference 1 and nonpatent literature 1 reference) Although 600-800 degrees C and the report which needs low temperature comparatively can also be seen on the other hand in the burning temperature of $LiNi1-XMnXO2$ which permuted $LiNiO2$ from Mn, it is the case that Mn substitutional rate is low strictly, and compounding the lithium content multiple oxide based on $LiNiO2$ at an elevated temperature 800 degrees C or more has a judgment that it is not so desirable. (For example, patent reference 2 reference)
[Patent reference 1]
JP,2002-42813,A official report (the four - 5th page, Fig. 10)
[Patent reference 2]
JP,8-171910,A official report (the 2nd page)
[0006] Column
[Nonpatent literature 1]
The collection 460-461 of 41 small ****** cell debate drafts (2000)
[Nonpatent literature 2]
E. Rossen et al. -- Solid State Ionics 57 (1992) 311-318
[0007]
[Problem(s) to be Solved by the Invention]
This invention offers the manufacture approach of the lithium-nickel-manganese compound by making $LiNi0.5Mn 0.5O2$ crystal completeness excelled [Mn] in output characteristics highly for the purpose of obtaining the positive active material for rechargeable lithium-ion batteries which has especially sufficient crystallinity and the outstanding electrochemistry property, and the ilmenite structure nickel-manganic acid ghost and lithium compound for obtaining said $LiNi0.5Mn 0.5O2$ react.
[0008]
As a result of this invention persons' considering correlation with the burning temperature of $LiNi0.5Mn 0.5O2$ which used the nickel-Mn compound hydroxide as the raw material, and compounded it, and the physical properties of said ingredient and an electrochemistry property, in the temperature region of 950 degrees C or more, migration of $nickel2+$ to Li layer, i.e., Cation mixing between 3a-3b sites, actualized, and it became clear that the output characteristics in heavy load conditions decline remarkably. Therefore, establishment of $LiNi0.5Mn 0.5O2$ which controlled said Cation mixing in $LiNi0.5Mn 0.5O2$ which used the nickel-Mn compound hydroxide as the raw material, and raised the output characteristics in a heavy load, and its manufacture approach was pressing need.
[0009]
Moreover, at the elevated temperature of 1000 degrees C, consumption of internal insulation is remarkable, and evapotranspiration of a lithium also becomes remarkable and tends to produce presentation variation. On the other hand, it compares with the thing of 800-900 degrees C which is seen by composition of conventional $LiCoO2$, $LiMn 2O4$, etc. comparatively calcinated at low temperature at elevated-temperature baking, and a manufacture top merit is large. Therefore, the manufacture approach compoundable at low temperature was expected $LiNi0.5Mn 0.5O2$ which maintains said crystal completeness and has the output characteristics of a heavy load.
[0010]
[Means for Solving the Problem]
In order to solve the above-mentioned technical problem the lithium-nickel-manganese multiple oxide of this invention General formula $Li1+XNi1/2+alphaMn1/2-alpha$ alpha value is set to $-0.05<=alpha<=0.05$, setting as $X>=0$ X value in the formula expressed with O2. The twin crystal particle or multiplex twin crystal particle which makes a twin plane the field in Miller indices hkl (104) to which the particle of said multiple oxide is hexagonal and belongs is contained. Diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) of

the powder X diffraction using Cu-K alpha rays is 1.15 or more. Moreover, the nickel of said lithium-nickel-manganese multiple oxide measured by the iodine titrimetric method and the average valence of manganese are characterized by or more 3.0 being 3.1 or less.

[0011]

Moreover, it is characterized by the manufacture approach consisting of a process which mixes an ilmenite mold nickel-manganic acid ghost and a lithium compound, and a process which calcinates said mixture above 750 degrees C under an owner oxygen ambient atmosphere.

[0012]

[Function]

Hereafter, this invention is explained to a detail.

[0013]

The lithium-nickel-manganese multiple oxide of this invention is expressed with general formula Li1+XNi1 / 2+alphaMn1 / 2-alphaO2. When it is X< 0 here, since there is an inclination which a byproduction phase generates and Cation mixing becomes remarkable, X>=0 is indispensable and is 0.01<=X<=0.10 preferably. If X value makes it increase too much, the rate of nickel3+ to nickel2+ leads [become high and] to the fall of electrochemistry capacity and is inconvenient. moreover, alpha value -0.05<=alpha<=0.05 -- it is alpha= 0 preferably. If alpha value exceeds 0.05, the rate of nickel3+ to nickel2+ will become high, and will affect a charge-and-discharge cycle property etc. Moreover, the single phase of stratified rock salt structure is not obtained as alpha is less than -0.05.

[0014]

Moreover, diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) of the powder X diffraction for which the lithium-nickel-manganese multiple oxide of this invention used Cu-K alpha rays is 1.15 or more. Said diffraction peak intensity ratio is the index of Cation mixing here, and when said intensity ratio is high, it is thought that the crystal completeness from which layer structure developed is high, and turbulence of the layer structure by Cation mixing exists when said intensity ratio is low (for example, Ohzuku et al., J.Elecrochem.Soc., 140, No.7, 1862 (1993)-1870). Said diffraction peak intensity ratio at which cation mixing does not actualize in this compound, but the output in a heavy load is maintained is 1.15 or more.

[0015]

Moreover, the average valence of the nickel and manganese by which the lithium-nickel-manganese multiple oxide of this invention was measured with the iodine titrimetric method is 3.1 or less [3.0 or more]. said average valence -- this -- it causes [generation of a byproduction phase actualizes that it is out of range, and] the fall of cell capacity and is not desirable.

[0016]

Furthermore, it became clear by FE-TEM and electron diffraction that it is the twin crystal particle or multiplex twin crystal particle which makes a twin plane the field in Miller indices hkl (104) which are hexagonal as for the lithium-nickel-manganese multiple oxide of this invention, and belong. Such structure is not peculiar to a stratified rock salt compound at large, and is characteristic of this invention.

[0017]

The manufacture approach of the positive active material for rechargeable lithium-ion batteries of this invention is characterized by consisting of a process which mixes an ilmenite structured type nickel manganic acid ghost and a lithium compound, and a process which calcinates said mixture above 750 degrees C under an owner oxygen ambient atmosphere. By these actuation, said positive-active-material Li1+XNi1 / 2+alphaMn1 said whose diffraction peak intensity ratio (003)/(104) is 1.15 or more / 2-alphaO2 is obtained. It is indispensable that the homogeneity in atomic level is maintained by the coprecipitation method, and the thing with high crystal completeness which has a good electrochemistry property is not obtained in actuation of powder alligation etc. Moreover, calcinating a nickel-Mn compound hydroxide below 750 degrees C or more 950 degrees C under an owner oxygen ambient atmosphere, when it is a raw material are the most important conditions that control Cation mixing. Under the present circumstances, a lithium compound can use a hydroxide, acetate, a carbonate, an oxalate, a nitrate, a chloride, a

sulfate, etc. In this case, it is desirable to use the hydroxide with which decomposition temperature does not generate corrosive gas low.

[0018]

Since the thing of ilmenite structure is high, its reactivity with a lithium compound is [the nickel manganic acid ghost used by this invention] desirable. An ilmenite structure nickel-manganic acid ghost is expressed with a chemical formula NiMnO3. Ilmenite structure is the ordered structure of corundum characteristic of FeTiO3, and it has the layer structure. A nickel/Mn atomic ratio has a desirable thing near 1, and, usually it becomes an oxygen deficiency mold. As the manufacture approach of the nickel-manganic acid ghost which has ilmenite structure, although the composition under high pressure can see mostly, composition by the ordinary pressure using the coprecipitation method using acetate and oxalic acid is also possible as shown in Journal of Alloys and Compounds and 196 75(1993)-79 for example.

[0019]

moreover, it is obtained even if it calcinates the nickel-manganese compound expressed with general formula nickel1/2+alphaMn1/2-alpha(OH)y(COO) z-nH2O (y+z=2, 0.5<=z<=1.5, and alpha -- the above -- the same -- n>=0) under an owner oxygen ambient atmosphere at 550 degrees C or more less than 800 degrees C.

[0020]

When nickel1/2+alphaMn1/2-alpha(OH)y(COO) z-nH2O is calcinated in airstream, ilmenite structure can obtain at 550 degrees C or more less than 800 degrees C.

[0021]

Moreover, an ilmenite structured type nickel-manganic acid ghost can be obtained also by calcinating a nickel-manganese compound hydroxide. As for a nickel-manganese compound hydroxide, it is desirable in that case that it is the uniform crystal phase to which nickel hydroxide and manganese hydroxide dissolved. Usually, it is possible to prepare with the hydroxide coprecipitation method which used the manganese salt of the nickel salt of NiSO4, nickel (NO3)2, and nickel(CH3COO) 2 grade and MnSO4 and Mn (NO3)2, and Mn(CH3COO) 2 grade as the raw material for preparing a nickel-manganese compound hydroxide. However, since the divalent manganese hydroxide of the obtained compound hydroxide is unstable, it is difficult to oxidize by the oxygen in air during actuation, to carry out the byproduction of the oxide of Mn3O4 grade, and to obtain the nickel-manganese compound hydroxide of a uniform crystal phase. Then, it is indispensable to oxidize the manganese in a nickel-manganese compound hydroxide in the condition more than trivalent [stable] with oxidizers, such as a hydrogen peroxide, while it has been in the uniform condition in which nickel hydroxide and manganese hydroxide dissolved, in order to prepare the nickel-manganese compound hydroxide of a crystal phase stable in air and uniform. In this case, the average oxidation number of nickel and manganese becomes 2.5 or more **s above nickel divalent and above manganese trivalent.

[0022]

It is thought that the oxidization by oxidizing agents, such as a hydrogen peroxide, is effective in oxidizing manganese hydroxide at a TOPOTA tick (structure not being changed) in the condition more than trivalent [like beta-oxy-manganese hydroxide (beta-MnOOH) / stable] unlike oxidization with the usual air etc. Moreover, since beta-oxy-manganese hydroxide has the crystal structure very near nickel hydroxide, it is considered that a compound hydroxide is stabilized that it is easy to maintain a uniform dissolution condition.

[0023]

Although especially the oxidation approach by the oxidizer is not limited, it can oxidize by, for example, feeding the nickel-manganese compound hydroxide obtained according to coprecipitation into the alkaline water solution which contained oxidizers, such as a hydrogen peroxide, as they were. Moreover, in case coprecipitation of the nickel-manganese compound hydroxide is carried out, complexing agents, such as ammonia which is added in order to raise the crystallinity of a nickel hydroxide, may be added. Thus, the nickel-manganic acid ghost which has ilmenite structure can be obtained by calcinating the obtained nickel-manganese compound under an owner oxygen ambient atmosphere. Under the present circumstances, although ilmenite structure is acquired in [about 300 degrees-C or more] less than 800 degrees C, the thing of

low crystallinity calcinated at the low temperature of 300-500 degrees C has reactivity highly desirable [structure] with a lithium compound.

[0024]

After dissolving lithium salt besides a dry-blending method in water or alcohol as the mixed approach of an ilmenite structured type nickel manganic acid ghost and a lithium compound and infiltrating a nickel-manganese compound, the approach using the wet slurry dried and calcinated is also effective. In addition, said slurry may be sprayed and dried by the spray. Moreover, the owner oxygen ambient atmosphere of a firing environments is indispensable. If it calcinates under anoxia ambient atmospheres, such as nitrogen and an argon, since reduction will arise, the target product is not obtained. An owner oxygen ambient atmosphere expresses the ambient atmosphere where oxygen tension is higher than air or air, such as pure oxygen, here.

[0025]

[Example]

Hereafter, this invention is explained in accordance with a concrete example.

[0026]

Example 1

After 450ml of water solutions which dissolved the 0.20 mols nickel sulfate and the 0.20-mol manganese sulfate, and 450ml of water solutions which dissolved the 0.80-mol sodium hydroxide were dropped at coincidence, performing stirring and nitrogen bubbling in 800ml of ammonium water solutions of 1.6 mol/L, night stirring mixing was carried out at 25 degrees C. Then, stirring pure-water 1.5L, the coprecipitation slurry of the ammonium water solution of 50ml of 1.6 mol/L, 100ml of 35% of hydrogen peroxide solution, and the obtained nickel-manganese was supplied in the above-mentioned sequence, and stirring was performed for further 3 hours. Then, filtration separation of the precipitate was carried out and it dried during a night at 80 degrees C.

[0027]

Thus, as a result of asking for the nickel-manganese average oxidation number of the obtained nickel-manganese compound hydroxide with an iodine titrimetric method, the average oxidation number was 2.71. Moreover, the X diffraction pattern was shown in drawing 1 . The peak of the single phase near nickel (OH)2 and beta-MnOOH was shown as drawing 1 . The account nickel of back to front-manganese compound hydroxide was calcinated among airstream at 400 degrees C for 2 hours. The presentation was expressed with NiMnO3 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed ilmenite structure as shown in drawing 2 .

[0028]

Moreover, said ilmenite structure nickel-manganic acid ghost and lithium-hydroxide monohydrate were mixed with the automatic mortar for 1 hour so that lithium atom/(nickel atom + manganese atom) (mole ratio) might be set to 1.01. The obtained mixture was calcinated at 750 degrees C among airstream for 12 hours, and the Li-nickel-Mn multiple oxide was obtained. It was Li1.01nickel0.50Mn 0.50O2 as a result of the ICP component analysis. R3m which an X diffraction pattern is as being shown in drawing 3 , and does not contain a byproduction phase, or its relative -- structure was shown. Diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) was 1.20 here.

[0029]

Average valence measurement by the iodine titrimetric method (nickel+Mn) was performed per said Li1.01nickel0.50Mn 0.50O2. The iodine titrimetric method performed DyerJ.American Chem Soc. and 76 (1954) 1499-1503 to reference.

[0030]

An iodine titrimetric method is the approach of using the property in which iodine separates quantitatively, if superfluous iodide ion is added to a strong oxidizer, and titrating and carrying out the quantum of this isolation iodine with a thiosulfate. After dissolving 0.3g of samples, and 3.0g of potassium iodide in the condition of having shaded to 6 N-HCl50cc, 1 N-NaOH200cc was added. After adding one cc of starch solutions to this, it titrated by 0.1 N-Na2S2O3. In addition, blank measurement by the system which does not contain a sample was also performed, and ****** was amended. Consequently, the average (nickel+Mn) valence was 3.054.

[0031]
Next, TEM observation was performed per said Li1.01nickel0.50Mn 0.5002. The sample made the micro sample what trickled and carried out the collection of the suspension which carried out ultrasonic distribution to the micro grid in ethanol, without grinding. JEOL JEM-2010F field emission mold 200 kV-TEM was used. The lattice image of a domain boundary was shown in drawing 4 . (003) The plaid corresponding to a spacing is making the include angle which is about 110 degrees, and the c-axis of two domains is making the include angle of about 70 degrees mutually. A domain boundary is not clear, two domains are mutually complex, and the part which lapped in the direction of incidence of an electron ray has appeared as moire-like crossover plaid. However, the domain boundary is almost parallel to a field (104). The ideal domain boundary model which makes a field a twin plane among drawing 4 (104) was shown. Thus, it is hexagonal, and the twin crystal particle or multiplex twin crystal particulate structure which makes a twin plane the field in belonging Miller indices hkl (104) is not peculiar to a stratified rock salt compound at large, and is characteristic of this invention.
[0032]
Furthermore, after mixing at a rate of 2:1 by the mixture (trade name: TAB-2) weight ratio of the polytetrafluoroethylene of an electric conduction agent, and acetylene black and casting this by the pressure of 1 ton/cm2 on a mesh (product made from SUS316) as a positive-electrode ingredient of a cell at a pellet type, reduced pressure drying was carried out at 150 degrees C, and the positive electrode for cells was produced. The cell was constituted using the obtained positive electrode for cells, the negative electrode which consists of a metal lithium foil (0.2mm in thickness), and the electrolytic solution which dissolved the 6 phosphorus-fluoride acid lithium in the mixed solvent of propylene carbonate and diethyl carbonate by the concentration of 1 mol/dm3. Thus, cell voltage carried out charge and discharge at 23 degrees C between 4.3V and 2.5V according to constant current using the produced cell. Charge was performed by 0.4 mA-cm -2, and two kinds of discharge was performed by the constant current of 0.4 mA-cm -2 and 2.5 mA-cm -2. The high rate discharge ratio was expressed with the discharge capacity factor (2.5 mA-cm-2 / 0.4 mA-cm -2). Consequently, they were initial discharge capacity (0.4 mA-cm -2) 147 mAh/g and 91.9% of high rate discharge ratios.
[0033]
Example 2
Moreover, the charge-and-discharge cycle trial per [which was compounded at 750 degrees C of example 1 publication] Li1.01nickel0.50Mn 0.5002 was performed up to 30 cycles by 0.4 mA-cm -2 like the example 1. The charge-and-discharge cycle property was shown in drawing 5 . From LiNi0.5Mn 0.5O2 obtained by the positive-electrode matter and the manufacture approach of this invention, most degradation was not seen by low-temperature baking of 750 degrees C, either.
[0034]
Example 3
Next, the nickel-manganese compound hydroxide was prepared like the example 1, and it calcinated among airstream at 650 degrees C for 2 hours. The presentation was expressed with NiMnO3 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide was compared with the example 1, and showed crystalline high ilmenite structure as it was shown in drawing 6 . Moreover, said ilmenite structure nickel-manganic acid ghost and lithium-hydroxide monohydrate were mixed with the automatic mortar for 1 hour so that lithium atom/(nickel atom + manganese atom) (mole ratio) might be set to 1.01. The obtained mixture was calcinated at 750 degrees C among airstream for 12 hours, and the Li-nickel-Mn multiple oxide was obtained. It was Li1.01nickel0.50Mn 0.5002 as a result of the ICP component analysis. As a result of performing the same cell evaluation trial as an example 1 per said Li1.01nickel0.50Mn 0.5002, initial discharge capacity (0.4 mA-cm -2) was 140.0 mAh/g, was compared with the example 1 and showed the low value. Therefore, in order to obtain LiNi0.5Mn 0.5O2 with the high cell engine performance, it is required for the crystallinity of ilmenite structure to be low, and, as for the burning temperature of a nickel-manganese compound hydroxide, it is desirable that it is 300-500 degrees C.

[0035]
Example 4
It prepared like the example 1 except having made burning temperature in lithiation into 900 degrees C. It was Li1.01nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 7 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.19 and the iodine titrimetric method (nickel+Mn), it was 3.072.
[0036]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 150.4 mAh/g and 89.9% of high rate discharge ratios.
[0037]
Example 5
It prepared like the example 1 except having made burning temperature in lithiation into 925 degrees C. It was Li1.01nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 8 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.21 and the iodine titrimetric method (nickel+Mn), it was 3.097.
[0038]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 147.0 mAh/g and 89.0% of high rate discharge ratios.
[0039]
Example 6
It prepared like the example 1 except having made burning temperature in lithiation into 950 degrees C. It was Li1.00nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 9 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.16 and the iodine titrimetric method (nickel+Mn), it was 3.087.
[0040]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 147.4 mAh/g and 87.5% of high rate discharge ratios.
[0041]
Example 7
A nickel-manganic acid ghost and lithium-hydroxide monohydrate were mixed so that lithium atom/(nickel atom + manganese atom) (mole ratio) might be set to 1.05, and it prepared like the example 1 except having made burning temperature in Li-izing into 900 degrees C. It was Li1.04nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 10 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.24 and the iodine titrimetric method (nickel+Mn), it was 3.080.
[0042]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 150.1 mAh/g and 89.2% of high rate discharge ratios.
[0043]
Example 8
A nickel-manganic acid ghost and lithium-hydroxide monohydrate were mixed so that lithium atom/(nickel atom + manganese atom) (mole ratio) might be set to 1.11, and it prepared like the example 1 except having made burning temperature in Li-izing into 900 degrees C. It was Li1.10nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 11 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003)

performed average valence measurement by 1.33 and the iodine titrimetric method (nickel+Mn), it was 3.091 here.
[0044]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 139.5 mAh/g and 94.8% of high rate discharge ratios.
[0045]
Example 9
Stirring mixing of the ammonium oxalate water-solution 1.0of 0.50-mol / L L was carried out for one day at addition and 25 degrees C at water-solution 1.0L which dissolved 0.50 mols nickel nitrate and 0.50-mol manganese nitrate. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. Thus, the presentation of the obtained precipitate was expressed with nickel0.49Mn0.51(OH) 1.0 (COO) 1.0.0.05H2O. The account precipitate of back to front was calcinated among airstream at 650 degrees C for 12 hours. The presentation was expressed with nickel0.49Mn 0.51O1.5 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed ilmenite structure.
[0046]
Moreover, after sinking a lithium-hydroxide water solution into said nickel0.49Mn 0.51O1.5, it calcinated at 1000 degrees C among airstream for 10 hours, and the Li-nickel-Mn multiple oxide was obtained. It was set to Li1.01nickel0.49Mn 0.51O2 as a result of ICP analysis. It is as the X diffraction pattern of said Li1.01nickel0.49Mn 0.51O2 being shown in drawing 12 . R3m which does not contain an impurity phase as drawing 12 , or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.18 and the iodine titrimetric method (nickel+Mn), it was 3.020.
[0047]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 140.4 mAh/g and 90.9% of high rate discharge ratios.
[0048]
Example 10
Stirring mixing of the ammonium oxalate water-solution 1.0L of 0.5 mol/L was carried out for one day at addition and 25 degrees C at mixed water solution 1.0L of the manganese nitrate of 0.5 mol/L, and the nickel nitrate of 0.5 mol/L. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. Thus, the presentation of the obtained precipitate was expressed with nickel0.50Mn0.50(OH) 1.0 (COO) 1.0.0.05H2O. Said precipitate was calcinated at 800 or 850 degrees C among [500, 550, 600, 650, 700, and 750] airstream for 12 hours, and the X diffraction pattern was investigated. Consequently, 550-degree-C or more range of the burning temperature of said precipitate with which ilmenite structure is acquired was less than 800 degrees C.
[0049]
Example 11
Stirring mixing of the ammonium oxalate water-solution 1.0of 0.50-mol / L L was carried out for one day at addition and 70 degrees C at water-solution 1.0L which dissolved the 0.50 mols nickel sulfate and the 0.50-mol manganese sulfate. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. Thus, the presentation of the obtained precipitate was expressed with nickel0.51Mn0.49(OH)1.0 (COO) 1.0. The account precipitate of back to front was calcinated among airstream at 650 degrees C for 12 hours. The presentation was expressed with nickel0.51Mn 0.49O1.5 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed the same ilmenite structure as drawing 6 .
[0050]
Moreover, the 1.0 mol/L acetic-acid lithium water solution was calcinated at 900 degrees C among the oxygen air current to it for 40 hours, after sinking into said nickel0.51Mn 0.49O1.5 so that lithium atom/(nickel atom + manganese atom) (mole ratio) may be set to 1.00, and the Li-nickel-Mn multiple oxide was obtained. R3m which is Li1.00nickel0.51Mn 0.49O2, and does not

contain the impurity phase as drawing 12 with the same X diffraction pattern as a result of ICP analysis, or its relative -- structure was shown. Moreover, as a result of performing the cell trial of example 1 publication, the initial discharge capacity of about 140 mAh/g was shown.
[0051]
Example 12
100ml of water solutions which dissolved 0.20-mol oxalic acid in 400ml of 4wt% acetic-acid solutions which dissolved 0.10 mols nickel acetate and 0.10-mol manganese acetate -- addition -- it was made to boil for 10 minutes and stirring mixing was carried out. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. Thus, the presentation of the obtained precipitate was expressed with nickel0.52Mn0.48(OH) 1.0 (COO) 1.0.0.07H2O. The account precipitate of back to front was calcinated among airstream at 650 degrees C for 12 hours. The presentation was expressed with nickel0.52Mn 0.48O1.5 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed the same ilmenite structure as drawing 6 .
[0052]
Moreover, the 1.0 mol/L acetic-acid lithium water solution was calcinated at 1050 degrees C among airstream to it for 5 hours, after sinking into said nickel0.52Mn 0.48O1.5 so that lithium atom/(nickel atom + manganese atom) (mole ratio) may be set to 1.05, and the Li-nickel-Mn multiple oxide was obtained. R3m which is Li1.05nickel0.52Mn 0.48O2, and does not contain the impurity phase as drawing 12 with the same X diffraction pattern as a result of ICP analysis, or its relative -- structure was shown. Moreover, as a result of performing the cell trial of example 1 publication, the initial discharge capacity of about 145 mAh/g was shown.
[0053]
Example 13
Stirring mixing of the ammonium oxalate water-solution 1.0L of 0.50 mol/L was carried out for one day at addition and 70 degrees C at water-solution 1.0L which dissolved 0.50 mols nickel acetate and 0.50-mol manganese acetate. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. Thus, the presentation of the obtained precipitate was expressed with nickel0.51Mn0.49(OH) 1.0 (COO) 1.0.0.08H2O. The account precipitate of back to front was calcinated among airstream at 650 degrees C for 12 hours. The presentation was expressed with nickel0.51Mn 0.49O1.5 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed the same ilmenite structure as drawing 6 .
[0054]
Moreover, the 1.0 mol/L acetic-acid lithium water solution was calcinated at 1000 degrees C among airstream to it for 12 hours, after sinking into said nickel0.51Mn 0.49O1.5 so that lithium atom/(nickel atom + manganese atom) (mole ratio) may be set to 1.00, and the Li-nickel-Mn multiple oxide was obtained. R3m which is Li1.00nickel0.51Mn 0.49O2, and does not contain the impurity phase as drawing 12 with the same X diffraction pattern as a result of ICP analysis, or its relative -- structure was shown. Moreover, as a result of performing the cell trial of example 1 publication, the initial discharge capacity of about 143 mAh/g was shown.
[0055]
Example 14
The nickel-manganese compound hydroxide obtained in the example 1 was calcinated among airstream at 600 degrees C for 3 hours. The presentation was expressed with NiMnO3 as a result of analyzing the obtained oxide chemically in ICP. Moreover, the X diffraction pattern of said oxide showed the same ilmenite structure as drawing 6 .
[0056]
Moreover, 1.0 mols / L lithium-hydroxide water solution was calcinated at 1000 degrees C among airstream to it for 10 hours, after sinking into said ilmenite structure nickel-manganic acid ghost so that lithium atom/(nickel atom + manganese atom) (mole ratio) may be set to 1.01, and the Li-nickel-Mn multiple oxide was obtained. R3m which is Li1.00nickel0.50Mn 0.50O2, and does not contain the byproduction phase as drawing 9 with the same X diffraction pattern as a result of ICP analysis, or its relative -- structure was shown. Moreover, as a result of performing the

cell trial of example 1 publication, the initial discharge capacity of about 154 mAh/g was shown.
[0057]
The example 1 of a comparison
It prepared like the example 1 except having made burning temperature in lithiation into 975 degrees C. It was Li1.01nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 13 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.10 and the iodine titrimetric method (nickel+Mn), it was 3.088.
[0058]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 139.8 mAh/g and 79.6% of high rate discharge ratios.
[0059]
The example 2 of a comparison
It prepared like the example 1 except having made burning temperature in lithiation into 1000 degrees C. It was Li1.01nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 14 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.06 and the iodine titrimetric method (nickel+Mn), it was 3.109.
[0060]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 133.9 mAh/g and 75.7% of high rate discharge ratios.
[0061]
The example 3 of a comparison
It prepared like the example 1 except having made burning temperature in lithiation into 1050 degrees C. It was Li1.00nickel0.50Mn 0.50O2 as a result of the ICP component analysis. as an X diffraction pattern being shown in drawing 15 -- it is -- R3m or its relative -- structure was shown. When diffraction peak intensity ratio (003)/(104) in the field and (104) field in Miller indices hkl (003) performed average valence measurement by 1.03 and the iodine titrimetric method (nickel+Mn), it was 3.098.
[0062]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 34.9 mAh/g and 33.0% of high rate discharge ratios.
[0063]
The example 4 of a comparison
A nickel-manganic acid ghost and lithium-hydroxide monohydrate were mixed so that lithium atom/(nickel atom + manganese atom) (mole ratio) might be set to 0.98, and it prepared like the example 1 except having made burning temperature in Li-izing into 900 degrees C. It was Li0.98nickel0.50Mn 0.50O2 as a result of the ICP component analysis. The mixed phase which an X diffraction pattern is as being shown in drawing 16 , and contains byproduction phases, such as Li2MnO3, was obtained. Moreover, it was 3.100 when average valence measurement by the iodine titrimetric method (nickel+Mn) was performed.
[0064]
Moreover, as a result of performing the same cell evaluation trial as an example 1, they were initial discharge capacity (0.4 mA-cm -2) 42.89 mAh/g and 23.8% of high rate discharge ratios.
[0065]
The example 5 of a comparison
Moreover, the charge-and-discharge cycle trial per [which was compounded at 1000 degrees C of example of comparison 2 publication] Li1.01nickel0.50Mn 0.50O2 was performed up to 30 cycles by 0.4 mA-cm -2 like the example 2. The charge-and-discharge cycle property was shown in drawing 17 . The inclination to compare with the charge-and-discharge cycle property Fig. of drawing 5 in an example 2, and to deteriorate was presented.
[0066]

The example 6 of a comparison
Stirring mixing of the ammonium oxalate water-solution 1.0of 0.50-mol / L L was carried out for one day at addition and 70 degrees C at water-solution 1.0L which dissolved 0.50 mols nickel nitrate and 0.50-mol manganese nitrate. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. The account precipitate of back to front was calcinated among airstream at 800 degrees C for 12 hours. As a result of analyzing the obtained oxide chemically in ICP, the Mn/nickel atomic ratio was 0.968. Moreover, the X diffraction pattern of said oxide showed Spinel structure.
[0067]
Moreover, after mixing said oxide and lithium-hydroxide monohydrate, it calcinated at 1000 degrees C among the oxygen air current for 10 hours, and the Li-nickel-Mn multiple oxide was obtained. the result of ICP analysis -- Li1.01nickel0.51Mn 0.49O2 -- it is -- an X diffraction pattern -- R3m or its relative -- structure was shown. However, as a result of performing the cell trial of example 1 publication, initial discharge capacity was about 125 mAh/g.
[0068]
The example 7 of a comparison
Stirring mixing of the ammonium oxalate water-solution 1.0of 0.50-mol / L L was carried out for one day at addition and 70 degrees C at water-solution 1.0L which dissolved 0.50 mols nickel nitrate and 0.50-mol manganese nitrate. Then, filtration separation of the precipitate was carried out and it dried for one day at 60 degrees C. The account precipitate of back to front was calcinated among airstream at 400 degrees C for 12 hours. As a result of analyzing the obtained oxide chemically in ICP, the Mn/nickel atomic ratio was 0.968. Moreover, the X diffraction pattern of said oxide showed the Spinel structure of low crystallinity.
[0069]
Moreover, after mixing said oxide and lithium-hydroxide monohydrate, it calcinated at 1000 degrees C among the oxygen air current for 10 hours, and the Li-nickel-Mn multiple oxide was obtained. R3m in which it is Li1.01nickel0.51Mn 0.49O2, and an X diffraction pattern does not contain an impurity phase as a result of ICP analysis, or its relative -- although structure was shown, as a result of performing the cell trial of example 1 publication, initial discharge capacity was about 115 mAh/g.
[0070]
The example 8 of a comparison
nickel0.49Mn 0.51O1.5 of ilmenite structure was compounded like the example 9.
[0071]
Moreover, after mixing said oxide and lithium-hydroxide monohydrate so that lithium atom/(nickel atom + manganese atom) (mole ratio) may be set to 1.05, it calcinated at 1100 degrees C among the oxygen air current for 10 hours, and the Li-nickel-Mn multiple oxide was obtained. the result of ICP analysis -- Li1.02nickel0.49Mn 0.51O2 -- it is -- an X diffraction pattern -- R3m or its relative -- structure was shown. However, the cell trial of example 1 publication showed a low initial discharge capacity of about 84 mAh/g.
[0072]
The example 9 of a comparison
nickel0.49Mn 0.51O1.5 of ilmenite structure was compounded like the example 9.
[0073]
Moreover, after mixing with said oxide and lithium-hydroxide monohydrate, it calcinated at 850 degrees C among the oxygen air current for 10 hours, and the Li-nickel-Mn multiple oxide was obtained. It was Li1.01nickel0.49Mn 0.51O2 as a result of ICP analysis. however, an X diffraction pattern -- R3m or its relative -- although structure was shown, it was low crystallinity comparatively. Moreover, as a result of performing the cell trial of example 1 publication, initial discharge capacity was about 99 mAh/g.
[0074]
X diffraction peak intensity ratio [of the example of this invention and the example of a comparison] (003)/(104) and a cell test result were shown in Table 1. A raw material A expresses the case where a raw material B uses nickel1/2+alphaMn1/2-alpha(OH)y(COO) z-

nH2O for a raw material here, when a nickel-Mn compound hydroxide is used for a raw material.
[0075]
Moreover, the average valence of the nickel in the lithium-nickel-manganese compound of this invention example and the example of a comparison and manganese was shown in Table 2.
[0076]
[Table 1]

実施例及び比較例	原料	焼成温度(℃)	Li/Metal原子比	X線回折ピーク強度比(003)/(104)	初期放電容量(mAh/g)	ハイレート放電比率(%)	備考
実施例1	A	750	1.01	1.20	147.0	91.9	本発明（750℃焼成）
実施例3	A	750	1.01	−	140.0	−	本発明（高結晶性イルメナイトを前駆体として使用）
実施例4	A	900	1.01	1.19	150.4	89.9	本発明（900℃焼成）
実施例5	A	925	1.02	1.21	147.0	89.0	本発明（925℃焼成）
実施例6	A	950	1.00	1.16	147.4	87.5	本発明（950℃焼成）
実施例7	A	900	1.04	1.24	150.1	89.2	本発明（Li/Me.比=1.04）
実施例8	A	900	1.10	1.33	139.5	94.8	本発明（Li/Me.比=1.10）
実施例9	B	1000	1.01	1.18	140.4	90.9	本発明（1000℃焼成）
比較例1	A	975	1.01	1.10	139.8	79.6	（975℃焼成）
比較例2	A	1000	1.01	1.06	133.9	75.9	（1000℃焼成）
比較例3	A	1050	1.00	1.03	34.90	33.0	（1050℃焼成）
比較例4	A	900	0.980	−	42.89	23.8	Li/Metal比範囲外（0.98）
比較例6	B	1000	1.01	−	125	−	Ni−Mnスピネル酸化物を使用
比較例7	B	1000	1.01	−	115	−	Ni−Mnスピネル酸化物を使用
比較例8	B	1100	1.02	−	84	−	（1100℃焼成）
比較例9	B	850	1.01	−	99	−	（850℃焼成）

[Table 2]

実施例及び比較例	焼成温度（℃）	Ｌｉ／Ｍｅｔａｌ原子比	（Ｎｉ＋Ｍｎ）平均価数
実施例１	７５０	１．０１	３．０５４
実施例４	９００	１．０１	３．０７２
実施例５	９２５	１．０２	３．０９７
実施例６	９５０	１．００	３．０８７
実施例７	９００	１．０４	３．０８０
実施例８	９００	１．１０	３．０９１
実施例９	１０００	１．０１	３．０２０
比較例１	９７５	１．０１	３．０８８
比較例２	１０００	１．０１	３．１０９
比較例３	１０５０	１．００	３．０９８
比較例４	９００	０．９８０	３．１００

[Effect of the Invention]
By using the lithium-nickel-manganese multiple oxide and its manufacture approach of this invention, the rechargeable lithium-ion battery using LiNi0.5+alphaMn 0.5-alphaO2 and this which controlled Cation mixing and raised the output characteristics in a heavy load can be offered.

[Brief Description of the Drawings]
[Drawing 1] The powder X diffraction Fig. of the nickel-manganese compound hydroxide obtained according to this invention example 1 is shown.
[Drawing 2] The powder X diffraction Fig. of NiMnO3 obtained according to this invention example 1 is shown.
[Drawing 3] The powder X diffraction Fig. of Li1.01nickel0.50Mn 0.50O2 obtained according to this invention example 1 is shown.
[Drawing 4] The TEM observation photograph and crystal structure model Fig. of Li1.01nickel0.50Mn 0.50O2 which were obtained according to this invention example 1 are shown.

[Drawing 5] The charge-and-discharge cycle property Fig. of Li1.02nickel0.50Mn 0.50O2 measured according to this invention example 2 is shown.
[Drawing 6] The powder X diffraction Fig. of NiMnO3 obtained according to this invention example 3 is shown.
[Drawing 7] The powder X diffraction Fig. of Li1.01nickel0.50Mn 0.50O2 obtained according to this invention example 4 is shown.
[Drawing 8] The powder X diffraction Fig. of Li1.01nickel0.50Mn 0.50O2 obtained according to this invention example 5 is shown.
[Drawing 9] The powder X diffraction Fig. of Li1.00nickel0.50Mn 0.50O2 obtained according to this invention example 6 is shown.
[Drawing 10] The powder X diffraction Fig. of Li1.04nickel0.50Mn 0.50O2 obtained according to this invention example 7 is shown.
[Drawing 11] The powder X diffraction Fig. of Li1.10nickel0.50Mn 0.50O2 obtained according to this invention example 8 is shown.
[Drawing 12] The powder X diffraction Fig. of Li1.01nickel0.49Mn 0.51O2 obtained according to this invention example 9 is shown.
[Drawing 13] The powder X diffraction Fig. of Li1.01nickel0.50Mn 0.50O2 obtained according to the example 1 of a comparison is shown.
[Drawing 14] The powder X diffraction Fig. of Li1.01nickel0.50Mn 0.50O2 obtained according to the example 2 of a comparison is shown.
[Drawing 15] The powder X diffraction Fig. of Li1.00nickel0.50Mn 0.50O2 obtained according to

the example 3 of a comparison is shown.
[Drawing 16] The powder X diffraction Fig. of Li0.98nickel0.50Mn 0.50O2 obtained according to the example 4 of a comparison is shown.
[Drawing 17] The charge-and-discharge cycle property Fig. of Li1.01nickel0.50Mn 0.50O2 measured according to the example 5 of a comparison is shown.

[Translation done.]